



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) Publication number:

0 298 222 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: **22.03.95** (51) Int. Cl.⁸: **C11D 3/395, C11D 17/00**

(21) Application number: **88107569.1**

(22) Date of filing: **11.05.88**

(54) **Encapsulated bleaches.**

(30) Priority: **10.07.87 US 71788**

(43) Date of publication of application:
11.01.89 Bulletin 89/02

(45) Publication of the grant of the patent:
22.03.95 Bulletin 95/12

(84) Designated Contracting States:
BE DE FR GB IT NL SE

(56) References cited:

EP-A- 0 070 474	AU-B- 496 577
US-A- 3 154 494	US-A- 3 322 674
US-A- 3 650 961	US-A- 3 703 470
US-A- 4 526 699	US-A- 4 657 784

(73) Proprietor: **ECOLAB INC.**
Ecolab Center
Saint Paul
Minnesota 55102 (US)

(72) Inventor: **Olson, Keith Edward**
13592 Eveleth Court
Apple Valley
Minnesota 55124 (US)

(74) Representative: **Maiwald, Walter, Dr.**
Dipl.-Chem. et al
Maiwald & Partner
Balanstrasse 57
D-81541 München (DE)

EP 0 298 222 B1

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

THIS PAGE BLANK (USPTO)

DescriptionField of the Invention

5 This invention relates to encapsulated bleaching agents having improved bleach stability in alkaline environments.

Background of the Invention

10 Bleaches are a well known group of chemical agents having the unique ability to remove color from a substrate without damaging the substrate. Because of this unique ability bleaches are often incorporated into cleaning compositions as a stain remover. However, most bleaching agents are unstable in typical cleaning compositions due to the alkaline conditions and/or the presence of free moisture.

Various attempts have been made to create a source of bleach which would be stable in cleaning
15 compositions including numerous attempts to encapsulate the bleach in various coating compounds.

Examples of such attempts are disclosed in U.S. Pat. 4,657,784, which is directed to a process for encapsulating a reactive core particle by at least two coatings to protect the particle from degradative interaction when contacted with reactive compounds, wherein the inner coating is a water-insoluble material and the outer coating must have a melting point higher than the melting point of the inner coating material
20 underneath.

European patent 0 070 474 is directed to a process for producing coated bleaching activators. These bleaching agents have a bleaching core consisting of N- or O-acyl compounds, preferably tetraacetylenediamine and a water-soluble cellulose ether, starch and starch ether as a single coating.

U.S. Pat. 3,650,961 is directed to a fluidized bed process for providing compositions composed of
25 particles concentrated within only one partially hydrated inorganic salt matrix or coating.

U.S. Pat. 4,526,699 is directed to bleaching compositions consisting of an N-halo compound with a coating comprising a silicate bound, hydrated, soluble inorganic salt and an alkali metal salt of boric acid by using a bleaching agent encapsulated by two water-soluble inorganic coatings.

Unfortunately, the encapsulated bleaches developed so far are either (i) substantially unstable in highly
30 alkaline environments such as found in solid cast detergents, (ii) difficult to manufacture, and/or (iii) prohibitively expensive to manufacture.

Accordingly, a substantial need exists for an inexpensive, easily manufactured source of bleach that is stable in a highly alkaline environment.

Summary of the Invention

I have discovered a source of bleach which can remain stable for extended periods of time in a highly alkaline environment. This source of bleach comprises an encapsulated bleach particle comprising a) a bleaching agent core, b) an inner coating and c) an outer coating, wherein the encapsulated bleach particle
40 is surrounded by two coatings and comprises an inner coating of a water-soluble separating compound in an amount sufficient to retard any chemical interaction between the bleaching agent core and an outer coating compound; an outer coating - provided directly on the inner coating - of an encapsulating amount of a water-soluble cellulose ether compound selected from the group consisting of (C₁₋₄) alkyl cellulose, carboxy (C₁₋₄) alkyl cellulose, hydroxy (C₁₋₄) alkyl cellulose, carboxy (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl
45 cellulose, (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl cellulose, and mixtures thereof.

I have discovered that while a bleach core can be effectively protected against an alkaline environment by a single coating of one of the listed water soluble cellulose ethers, the cellulose ether can itself, under the proper conditions, react with and deactivate the bleach core. Accordingly, I have found it preferable to employ an inner coating of a chemically compatible compound to separate the bleach core from the
50 cellulose ether outer coating.

As utilized herein, including the claims, "inner coating" refers to that coating layer in physical contact with the core material.

Detailed Description of the Invention Including a Best Mode

55 My stable bleaching composition comprises a bleach core encapsulated in an inner coating of a bleach compatible separating compound and an outer coating of a water soluble cellulose ether.

BLEACHING AGENT

Bleaches suitable for use as the core component include any of the well known bleaching agents capable of removing stains from such substrates as dishes, flatware, pots and pans, textiles, countertops, appliances, flooring, etc. without significantly damaging the substrate. A nonlimiting list of such bleaches includes active halogen releasing bleaches such as hypochlorites, chlorites, chlorinated phosphates, chloroisocyanates and chloroamines; and peroxide compounds such as hydrogen peroxide, perborates and percarbonates. Preferred bleaches include those bleaches which liberate an active halogen species such as Cl^- , Br^- , OCl^- , or OBr^- under conditions normally encountered in typical cleaning processes. Most preferably, the bleaching agent releases Cl^- or OCl^- . A nonlimiting list of useful chlorine releasing bleaches includes calcium hypochlorite, lithium hypochlorite, chlorinated trisodium phosphate, sodium dichloroisocyanurate, potassium dichloroisocyanurate, [(monotrichloro)-tetra(monopotassiumdichloro)] pentaisocyanurate, monochloroamine, dichloroamine, trichloromelamine, sulfondichloro-amide, 1,3-dichloro-5,5-dimethyl hydantoin, N-chloroammeline, N-chlorosuccinimide, N,N'-dichloroazodicarbonimide, N,N-chloroacetyl urea, N,N'-dichlorobiuret, chlorinated dicyanamide, trichlorocyanuric acid, and hydrates thereof.

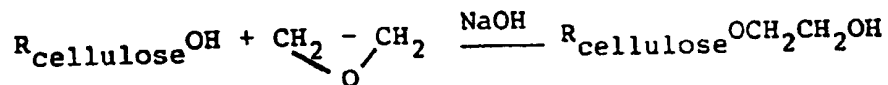
Because of their low cost and high bleaching efficiency the most preferred bleaching agents are the alkali metal salts of chloroisocyanurates and the hydrates thereof.

SEPARATING COMPOUNDS

Compounds suitable for use as the inner coating component include any compound which is solid at those temperatures likely to be encountered during storage of the encapsulated bleach (i.e. -5° to 50°C), is chemically compatible with (i.e. does not react with) either the bleaching agent core or the water soluble cellulose ether outer coating, and is capable of separating the bleaching agent from the cellulose ether so as to prevent deactivation of the bleach by the cellulose ether. Useful separating compounds include specifically but not exclusively water insoluble compounds such as C_{11} - 30 fatty acids, waxes and water soluble compounds such as alkyl sulfonates, detergent builders and detergent fillers. Because of their ability to readily release the bleach core under conditions typically encountered during detergent use, the water soluble compounds are preferred. Most preferably, the separating compound is an inorganic detergent builder or filler useful in the cleaning composition into which the bleach is to be employed. A nonlimiting list of such detergent builders and fillers includes inorganic compounds such as sodium sulfate, sodium chloride, tetrasodium pyrophosphate, alkali metal silicates, tetrapotassium pyrophosphate, pentasodium tripolyphosphate, pentapotassium tripolyphosphate, sodium sequeicarbonate, potassium sequeicarbonate and phytates. Because of their low cost, ease of availability, ease of use and efficient detergent building properties the inner coating compound preferably comprises a mixture of sodium sulfate and a tripolyphosphate.

WATER SOLUBLE CELLULOSE ETHERS

Cellulose is a linear polymer of anhydroglucose units held together by glucosidic linkages. Each anhydroglucose unit contains three hydroxyl groups - one primary and two secondary. Cellulose derivatives such as cellulose ethers are formed by reaction of the cellulose with a chemical reagent at these hydroxyl groups. For example, hydroxyethylcellulose can be prepared by the reaction of alkali cellulose with ethylene oxide in the presence of isopropanol, tert-butanol or acetone in accordance with the following equation:

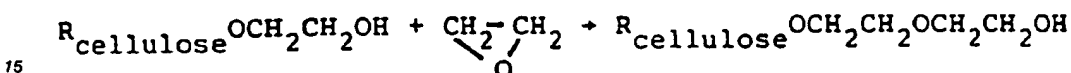
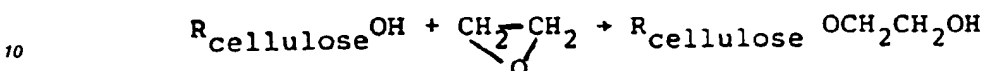


Cellulose derivatives useful as the outer coating component in the present invention are the water soluble cellulose ethers selected from the group consisting of (C_1-4) alkyl cellulose, carboxy (C_1-4) alkyl cellulose, hydroxy (C_1-4) alkyl cellulose di (C_1-4) alkyl carboxy (C_1-4) hydroxy (C_1-4) cellulose, (C_1-4) alkyl hydroxy (C_1-4) alkyl cellulose and mixtures thereof. For reasons of superior bleach stabilizing performance and ease of application, the preferred cellulose ethers are the hydroxy (C_1-4) alkyl celluloses with the most preferred cellulose ethers being hydroxyethylcellulose and hydroxy-propylcellulose.

In most commercially available cellulose derivatives, some of the hydroxyl groups are not substituted. The number of unsubstituted hydroxyl groups is known as the degree of substitution (DS) and is designated

by a number from 0 to 3 which represents the average number of hydroxyl groups, of the three available in the anhydroglucose unit, that have been substituted.

A special problem arises in the expression of degree of substitution for hydroxyalkyl derivatives because each time a hydroxyalkyl substituent is added, a new reactive hydroxyl group is formed and the number of reactive hydroxyl sites does not change. The result is the formation of side chains, as shown below:



To describe the extent of the formation of side chains the term MS has been coined. MS is defined as the number of moles of reagent (i.e. ethylene oxide) combined per anhydroglucose unit.

The ratio of DS to MS is an indication of the average length of the side chains developed. The DS, MS and ratio of DS to MS can affect the chemical properties of the cellulose derivative and only those cellulose ethers that have a DS, MS and DS:MS which result in a water soluble compound may be usefully employed in the present invention.

The DS of several useful cellulose ethers are set forth below:

Table 1

Cellulose	Typical DS	Preferred DS
Hydroxymethyl	0-2.6	1.3-2.6
Hydroxyethyl	0-3	1.2-3
Hydroxypropyl	1.4-3	1.4-3
Carboxymethyl	0.4-1.4	0.7-0.9

The composition can comprise 20 to 90 wt-%, preferably 40 to 70 wt-% bleach core, 5 to 60 wt-%, preferably 10 to 50 wt-% separating compound inner coating and 1 to 25 wt-%, preferably 2 to 10 wt-% water soluble cellulose ether outer coating.

While not intending to be limited thereby I believe that the water soluble cellulose ethers described herein are capable of protecting a bleaching agent core from deactivation in an alkaline environment because the cellulose ethers are water insoluble when in the presence of at least 10-50 wt-% inorganic salts such as sodium chloride, sodium sulphate and sodium perborate (i.e. those conditions typically encountered in solid detergents) and water soluble only when the wt-% of inorganic salt falls outside these levels (i.e. those conditions typically encountered during use of the detergent).

ENCAPSULATION PROCEDURE

The bleach may be encapsulated in any convenient manner capable of ensuring complete coating of the bleach. Obtaining a complete protective coating with the cellulose ether is simplified by the tendency of cellulose ethers to naturally form a nonporous, evenly distributed coating on a particle. For reasons of low manufacturing cost and ease of manufacture the bleach is preferably encapsulated in a fluidized bed as set forth in detail in the Examples. Briefly, the separating composition is dissolved in an appropriate solvent, such as water when water soluble, to form an inner coating solution; the water soluble cellulose ether dissolved in water to form an outer coating solution; the bleach particles fluidized in a fluidized bed apparatus, the inner coating solution sprayed onto the fluidized particles and dried, and the outer coating solution sprayed on the fluidized particles and dried.

Example I

Into a 32 liter container was placed 5.96 Kg granular sodium sulfate, 1.62 Kg sodium tripolyphosphate and 23.78 Kg water to form a first coating solution.

5 Into a fluidized bed was placed 14.59 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56® was fluidized with air and the bed heated to 68-74° C. The entire amount of first coating solution was sprayed onto the CDB-56® granules through a Gustav Schlick Nozzle Model 941, at an atomization air pressure of $275.8 \cdot 10^3$ Pa (40 psig), to form once coated CDB-56® particles.

10 Into the now empty 32 liter container was placed 1.14 Kg KLUCEL J®, a hydroxypropylcellulose purchased from Hercules, Inc., and 34.47 Kg water to form a second coating solution. The bed temperature was adjusted to 71-72° C. and the entire amount of second coating solution sprayed onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice coated, protectively encapsulated CDB-56 particles. The bed temperature was then adjusted to 74° C. and the protectively encapsulated CDB-56® particles dried. The process yielded 23.15 Kg of protectively encapsulated CDB-56® particles comprising 15 60 wt-% core of CDB-56®, 35 wt-% first coat of a mixture of 75 wt-% sodium sulfate and 25 wt-% sodium tripolyphosphate hexahydrate and 5 wt-% second coat of KLUCEL J®.

Example II

20 Into a 32 liter container was placed 5.96 Kg granular sodium sulfate, 1.62 Kg sodium tripolyphosphate and 23.78 Kg water to form a first coating solution.

Into a fluidized bed was placed 13.43 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56® was fluidized with air and the 25 bed heated to 72-74° C. The entire amount of first coating solution was sprayed onto the CDB-56® granules through a Gustav Schlick Nozzle Model, 941 at an atomized air pressure of $275.8 \cdot 10^3$ Pa (40 psig), to form once CDB-56® coated particles.

Into the now empty 32 liter container was placed 2.27 Kg KLUCEL J®, a hydroxypropylcellulose purchased from Hercules, Inc., and 70.94 Kg water to form a second coating solution. The bed temperature 30 was adjusted to 69-71° C. and the entire amount of second coating solution sprayed onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice-coated, protectively encapsulated CDB-56® particles. The bed temperature was then adjusted to 74° C. and the protectively encapsulated CDB-56® particles dried. The process yielded 20.14 Kg of protectively encapsulated CDB-56® particles comprising 55 wt-% core of CDB-56®, 35 wt-% first coat of a mixture of 75 wt-% sodium sulfate and 25 wt-% 35 sodium tripolyphosphate hexahydrate and 10 wt-% second coat of KLUCEL J®.

Example III

40 Into a 32 liter container was placed 7.26 Kg sodium sulfate, 2.42 Kg sodium tripolyphosphate and 30.36 Kg water to form a first coating solution.

Into a fluidized bed was placed 12.25 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56 was fluidized with air and the bed heated to 63-71° C. The entire amount of first coating solution was sprayed onto the CDB-56® granules through a Gustav Schlick Nozzle Model 941, at an atomized air pressure of $275.8 \cdot 10^3$ Pa (40 psig), to form 45 once coated CDB-56® particles.

Into the now empty 32 liter container was placed 2.27 Kg KLUCEL J®, a hydroxypropylcellulose purchased from Hercules, Inc., and 70.94 Kg water to form a second coating solution. The bed temperature was adjusted to 69-71° C. and the entire amount of second coating solution sprayed onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice-coated, protectively encapsulated CDB-56® particles. The bed temperature was then adjusted to 74° C. and the protectively encapsulated CDB-56® particles dried. The process yielded 21.91 Kg of protectively encapsulated CDB-56® particles 50 comprising 50 wt-% core of CDB-56, 45 wt-% first coat of a mixture of 71 wt-% sodium sulfate and 29 wt-% sodium tripolyphosphate hexahydrate and 5 wt-% second coat of KLUCEL J®.

55 Example IV

Into a 32 liter container was placed 2.38 Kg granular sodium sulfate, 0.79 Kg sodium tripolyphosphate hexahydrate and 9.50 Kg water to form a first coating solution.

Into a fluidized bed was placed 5.83 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56® was fluidized with air heated to 61 ° C. The entire amount of first coating solution was sprayed over the spray period onto the CDB-56® granules through a Gustav Schlick Nozzle Model 941 to at an atomization air pressure of $206.8 \cdot 10^3$ Pa (30 psi) to form once coated CDB-56® particles.

Into the now empty 32 liter container was placed 0.45 Kg of a blend of 66 Wt-% Lr Natrosol 250® and 34 Wt-% Natrosol 250®, both of which are hydroxyethylcelluloses purchased from Hercules, Inc., and 22.7 Kg water to form a second coating solution. The bed temperature was adjusted to an average of 70 ° C. and the entire amount of second coating solution sprayed over the spray period onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice coated, protectively encapsulated CDB-56® particles. The bed temperature was then adjusted to 74 ° C. and the protectively encapsulated CDB-56® particles dried. The process yielded 8.89 Kg of protectively encapsulated CDB-56® particles comprising 60 wt-% core of CDB-56®, 35 wt-% first coat of a mixture of 75 wt-% sodium sulfate and 25 wt-% sodium tripolyphosphate hexahydrate and 5 wt-% second coat of hydroxyethylcellulose.

Example V

Into a 32 liter container was placed 2.38 Kg granular sodium sulfate, 0.79 Kg sodium tripolyphosphate hexahydrate and 9.5 Kg water to form a first coating solution.

Into a fluidized bed was placed 5.83 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56® was fluidized with air heated to an average of 62 ° C. The entire amount of first coating solution was sprayed over the spray period onto the CDB-56® granules through a Gustav Schlick Nozzle Model 941, at an atomization air pressure of 30 psi to form once CDB-56® coated particles.

Into the now empty 32 liter container was placed 0.45 Kg Methocel® type F4M, a hydroxypropylmethylcellulose, a methylcellulose purchased from Dow Chemical, Inc., and 22.7 Kg water to form a second coating solution. The bed temperature was adjusted to an average of 71 ° C. and the entire amount of second coating solution sprayed over the spray period onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice-coated, protectively encapsulated CDB-56® particles. The protectively encapsulated CDB-56® particles were then dried. The process yielded 8.87 Kg of protectively encapsulated CDB-56® particles comprising 60 wt-% core of CDB-56®, 35 wt-% first coat of a mixture of 75 wt-% sodium sulfate and 25 wt-% sodium tripolyphosphate hexahydrate and 5 wt-% second coat of hydroxypropylmethylcellulose.

Example VI

Into a 32 liter container was placed 2.38 Kg granular sodium sulfate, 2.38 Kg sodium tripolyphosphate hexahydrate and 9.5 Kg water to form a first coating solution.

Into a fluidized bed was placed 5.83 Kg CDB-56®, a granular dichloroisocyanurate dihydrate purchased from FMC and now available from Olin Corporation. The CDB-56® was fluidized with air heated to 65 ° C. The entire amount of first coating solution was sprayed over the spray period onto the CDB-56® granules through a Gustav Schlick Nozzle Model 941, at an atomization air pressure of $206.8 \cdot 10^3$ Pa (30 psi) to form once coated CDB-56® particles.

Into the now empty 32 liter container was placed 4.5 Kg CMC-CLT®, a sodium carboxymethylcellulose purchased from Hercules, Inc., and 22.7 Kg water to form a second coating solution. The bed temperature was adjusted to an average of 71 ° C. and the entire amount of second coating solution sprayed over the spray period onto the once coated CDB-56® particles through the Gustav Schlick nozzle to form twice-coated, protectively encapsulated CDB-56® particles. The protectively encapsulated CDB-56® particles were dried. The process yielded 8.98 Kg of protectively encapsulated CDB-56® particles comprising 60 wt-% core of CDB-56®, 35 wt-% first coat of a mixture of 75 wt-% sodium sulfate and 25 wt-% sodium tripolyphosphate hexahydrate and 5 wt-% second coat of sodium carboxymethyl cellulose.

Example VII

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially demineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents of the reaction vessel were heated to an average temperature of 77 ° C. and held at that temperature for 70 minutes to form hydrated metasilicate. The heating means was then removed from the reaction vessel and

the temperature of the hydrated metasilicate allowed to fall below 65° C. A premix of 2.2 grams of mono and di alkyl acid phosphate esters rich in C₁₆, 13.8 grams of nonionic ethylene propylene oxide block copolymers terminated in propylene oxide and 399.4 grams of hydrated sodium tripolyphosphate containing 19.4 wt-% water of hydration was added to the hydrated metasilicate to form a slurry. The slurry was then
 5 thoroughly mixed and cooled to 56° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example I. The contents of the container were quickly agitated for about 10 seconds and then solidified by cooling.

The percent active chlorine remaining in the composition after storage at 37.7° C. (100° Fahrenheit) for 2 and 4 weeks was titrationally determined to be 88.4 and 90.0% respectively.

10

Example VIII

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially dimineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents
 15 of the laboratory beaker were heated to an average temperature of 78° C. and held at that temperature for 69 minutes to form hydrated metasilicate. The heat source was then removed from the reaction vessel and the temperature of the hydrated metasilicate allowed to fall below 66° C. A premix of 2.2 grams of mono and dialkyl acid phosphate ester rich in C₁₆, 13.8 grams of nonionic ethylene propylene oxide block copolymers terminated in propylene oxide and 399.4 grams hydrated sodium tripolyphosphate containing
 20 19.4 wt-% water of hydration was added to the hydrated metasilicate to form a slurry. This slurry was then thoroughly mixed and cooled to 53° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example II. The contents of the container were quickly agitated for about 10 seconds and then solidified by cooling. The percent active chlorine remaining in the composition after storage at 37.7° C. (100° F) for 2 and 4 weeks
 25 was titrationally determined to be 82.2% and 84.5% respectively.

Example IX

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially dimineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents
 30 of the reaction vessel were heated to an average temperature of 78° C. and held at that temperature for 57 minutes to form hydrated metasilicate. The heat source was then removed from the reaction vessel and the temperature of the hydrated metasilicate allowed to fall below 66° C. A premix of 2.2 grams of mono and dialkyl acid phosphate esters rich in C₁₆, 13.8 grams of nonionic ethylene propylene oxide block
 35 copolymers terminated in propylene oxide and 399.4 grams of hydrated sodium tripolyphosphate containing 19.4 wt-% water of hydration was added to the hydrated metasilicate to form a slurry. This slurry was then thoroughly mixed and cooled to 52° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example III. The contents of the container were quickly agitated for about 10 seconds and then solidified by cooling.
 40 The percent active chlorine remaining in the composition after storage at 37.7° C. (100° F.) for 2 and 4 weeks was titrationally determined to be 89.4% and 89.2% respectively.

Example X

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially dimineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents
 45 of the reaction vessel were heated to an average temperature of 86° C. and held at that temperature for 80 minutes to form hydrated metasilicate. The heating means was then removed from the reaction vessel and the temperature of the hydrated metasilicate allowed to fall below 63° C. A premix of 2.3 grams of mono and dialkyl acid phosphate esters rich in C₁₆, 13.9 grams of nonionic ethylene propylene oxide block
 50 copolymers terminated in propylene oxide and 399.2 grams hydrated sodium tripolyphosphate containing 19.4 wt% water was added to the hydrated metasilicate to form a slurry. This slurry was then thoroughly mixed and cooled to 56° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example IV. The
 55 contents of the container were quickly agitated for about 10 seconds and then solidified by cooling.

The percent active chlorine remaining in the composition after storage at 37.7° C. (100° F) for 2 and 4 weeks was titrationally determined to be 91.5% and 84.6% respectively.

Example XI

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially demineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents of the reaction vessel were heated to an average temperature of 73° C. and held at that temperature for 62 minutes to form hydrated metasilicate. The heat source was then removed from the reaction vessel and the temperature of the hydrated metasilicate allowed to fall below 61° C. A premix of 2.3 grams of mono and dialkyl acid phosphate ester rich in C₁₆, 13.8 grams of nonionic ethylene propylene oxide block copolymers terminated in propylene oxide and 399.2 grams hydrated sodium tripolyphosphate containing 19.4 wt-% water of hydration was added to the hydrated metasilicate to form a slurry. This slurry was then thoroughly mixed and cooled to 50° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example V. The contents of the container were quickly agitated for about 10 seconds and then solidified by cooling.

The percent active chlorine remaining in the composition after storage at 37.7° C. (100° F) for 2 weeks was titrationally determined to be 84.1%.

Example XII

Into a laboratory beaker, equipped with a stirring means and a heating means, was placed 234.9 grams of substantially demineralized water followed by 356.7 grams anhydrous sodium metasilicate. The contents of the reaction vessel were heated to an average temperature of 77° C. and held at that temperature for 65 minutes to form hydrated metasilicate. The heat source was then removed from the reaction vessel and the temperature of the hydrated metasilicate allowed to fall below 60° C. A premix of 2.3 grams of mono and dialkyl acid phosphate ester rich in C₁₆, 13.9 grams of nonionic ethylene propylene oxide block copolymers terminated in propylene oxide and 399.2 grams hydrated sodium tripolyphosphate containing 19.4 wt-% water of hydration was added to the hydrated metasilicate to form a slurry. This slurry was then thoroughly mixed and cooled to 50° C. 97.5 grams of the slurry was then poured into a 0.1 liter container simultaneously with 2.5 grams of the encapsulated bleach made in accordance with Example VI. The contents of the container were quickly agitated for about 10 seconds and then solidified by cooling.

The percent active chlorine remaining in the composition after storage at 37.7° C. (100° F) for 2 weeks was titrationally determined to be 92%.

Claims

1. An encapsulated bleach particle comprising
 - a) a bleaching agent core
 - b) an inner coating
 - c) an outer coating
 characterized in that the encapsulated bleach particle is surrounded by two coatings and comprises
 - an inner coating of a water-soluble separating compound in an amount sufficient to retard any chemical inter-action between the bleaching agent core and an outer coating compound;
 - an outer coating - provided directly on the inner coating - of an encapsulating amount of a water-soluble cellulose ether compound selected from the group consisting of (C₁₋₄) alkyl cellulose, carboxy (C₁₋₄) alkyl cellulose, hydroxy (C₁₋₄) alkyl cellulose, carboxy (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl cellulose, (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl cellulose, and mixtures thereof.
2. The encapsulated particle of claim 1 wherein the bleaching agent is a source of active halogen.
3. The encapsulated particle of claim 2 wherein the bleaching agent is an alkali metal dichloroisocyanurate, and hydrates thereof.
4. The encapsulated particle of claim 1 wherein the separating compound is a water-soluble detergent builder or filler.
5. The encapsulated particle of claim 4 wherein the detergent builder or filler is sodium sulfate, sodium chloride, a condensed phosphate or a combination thereof.

6. The encapsulated particle of claim 5 wherein the water-soluble cellulose ether is a hydroxy (C₁₋₄) alkyl cellulose.
7. The encapsulated particle of claim 6 wherein the hydroxy (C₁₋₄) alkyl cellulose is hydroxypropyl cellulose or hydroxyethyl cellulose.
8. The encapsulated particle of claim 7 wherein the hydroxy (C₁₋₄) alkyl cellulose has a DS of 0.7 to 3.0.
9. The encapsulated particle of claim 8 wherein the hydroxypropyl cellulose has a DS of 1.4 to 3.0.
10. The encapsulated particle of claim 8 wherein the hydroxyethyl cellulose has a DS of 1.2 to 3.0.
11. The encapsulated particle of claim 1 wherein the encapsulated particle comprises 40 to 70 wt-% core, 10 to 50 wt-% water-soluble inner coating compound and 2 to 10 wt-% outer coating compound.
12. An encapsulated bleach particle according to claim 1, comprising:
 - (a) 20 to 90 wt-% core of an active chlorine source;
 - (b) 5 to 60 wt-% water-soluble inner coating of a detergent builder or filler surrounding and in physical contact with the core; and
 - (c) 1 to 25 wt-% outer encapsulating coating of a hydroxy (C₁₋₄) alkyl cellulose which is physically separated from the core of the active chlorine source by the inner coating.
13. The particle of claim 12 wherein the particle comprises:
 - (a) 40 to 70 wt-% core;
 - (b) 10 to 50 wt-% water-soluble inner coating of sodium sulfate, sodium chloride, a condensed phosphate or a combination thereof; and
 - (c) 2 to 10 wt-% outer coating of hydroxypropyl cellulose or hydroxyethyl cellulose.

Patentansprüche

1. Einkapseltes Bleichmittelteilchen umfassend
 - a) einen Bleichmittelkern
 - b) eine innere Beschichtung
 - c) eine äußere Beschichtungdadurch gekennzeichnet, daß das eingekapselte Bleichmittelteilchen von zwei Beschichtungen umschlossen ist, und eine innere Beschichtung aus einer wasserlöslichen Trennverbindung, in einer Menge, die ausreichend ist, um jegliche chemische Wechselwirkung zwischen dem Bleichmittelkern und einer äußeren Beschichtungsverbindung zu verlangsamen;
eine direkt auf der inneren Beschichtung vorgesehene äußere Beschichtung aus einem einkapselnden Gehalt an einer wasserlöslichen Celluloseetherverbindung, ausgewählt aus der Gruppe, die aus (C₁₋₄)-Alkylcellulose, Carboxy-(C₁₋₄)-Alkylcellulose, Hydroxy-(C₁₋₄)-Alkylcellulose, Carboxy-(C₁₋₄)-Alkylhydroxy-(C₁₋₄)-Alkylcellulose, (C₁₋₄)-Alkylhydroxy-(C₁₋₄)-Alkylcellulose und Mischungen dieser besteht;
umfaßt.
2. Einkapseltes Teilchen nach Anspruch 1, wobei das Bleichmittel eine Aktivhalogenquelle ist.
3. Einkapseltes Teilchen nach Anspruch 2, wobei das Bleichmittel ein Alkalidichloroisocyanurat oder dessen Hydrat ist.
4. Einkapseltes Teilchen nach Anspruch 1, wobei die Trennverbindung ein wasserlöslicher Builder oder Füllstoff ist.
5. Einkapseltes Teilchen nach Anspruch 4, wobei der Builder oder Füllstoff Natriumsulfat, Natriumchlorid, ein kondensiertes Phosphat oder eine Kombination dieser ist.

6. Einkapseltes Teilchen nach Anspruch 5, wobei der wasserlösliche Celluloseether eine Hydroxy-(C₁₋₄)-Alkylcellulose ist.
7. Einkapseltes Teilchen nach Anspruch 6, wobei die Hydroxy-(C₁₋₄)-Alkylcellulose Hydroxypropylcellulose oder Hydroxyethylcellulose ist.
8. Einkapseltes Teilchen nach Anspruch 7, wobei die Hydroxy-(C₁₋₄)-Alkylcellulose einen Substitutionsgrad von 0,7 bis 3,0 hat.
9. Einkapseltes Teilchen nach Anspruch 8, wobei die Hydroxypropylcellulose einen Substitutionsgrad von 1,4 bis 3,0 hat.
10. Einkapseltes Teilchen nach Anspruch 8, wobei die Hydroxyethylcellulose einen Substitutionsgrad von 1,2 bis 3,0 hat.
11. Einkapseltes Teilchen nach Anspruch 1, wobei das eingekapselte Teilchen 40 bis 70 Gew.-% Kern, 10 bis 50 Gew.-% wasserlösliche innere Beschichtungsverbindung und 2 bis 10 Gew.-% äußere Beschichtungsverbindung umfaßt.
12. Einkapseltes Bleichmittelteilchen gemäß Anspruch 1, umfassend:
 - a) 20 bis 90 Gew.-% Kern aus einer Aktivchlorquelle;
 - b) 5 bis 60 Gew.-% wasserlösliche innere Beschichtung aus einem Builder oder Füllstoff, die in physischem Kontakt mit dem Kern steht und diesen umschließt; und
 - c) 1 bis 25 Gew.-% äußere einkapselnde Beschichtung aus einer Hydroxy-(C₁₋₄)-Alkylcellulose, die durch die innere Beschichtung physisch von dem Kern aus der Aktivchlorquelle getrennt ist.
13. Teilchen nach Anspruch 12, wobei das Teilchen umfaßt:
 - a) 40 bis 70 Gew.-% Kern;
 - b) 10 bis 50 Gew.-% wasserlösliche innere Beschichtung aus Natriumsulfat, Natriumchlorid, einem kondensiertem Phosphat oder einer Kombination dieser; und
 - c) 2 bis 10 Gew.-% äußere Beschichtung aus Hydroxypropylcellulose oder Hydroxyethylcellulose.

Revendications

1. Particule de blanchiment encapsulée comprenant :
 - a) un noyau de blanchiment,
 - b) un revêtement intérieur,
 - c) un revêtement extérieur,caractérisée en ce que la particule de blanchiment encapsulée est enrobée de deux revêtements et comprend un revêtement intérieur constitué par un composé de séparation soluble dans l'eau en quantité suffisante pour retarder toute interaction chimique entre le noyau d'agent de blanchiment et un revêtement extérieur; un revêtement extérieur - prévu directement sur le revêtement intérieur - constitué par une quantité encapsulante d'éther cellulose soluble dans l'eau et choisie dans le groupe constitué par les (C₁₋₄) alkyl celluloses, les carboxy (C₁₋₄) alkyl celluloses, les hydroxy (C₁₋₄) alkyl celluloses, les carboxy (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl celluloses, les (C₁₋₄) alkyl hydroxy (C₁₋₄) alkyl celluloses et les mélanges de ces composés.
2. Particule encapsulée selon la revendication 1 dans laquelle l'agent de blanchiment est une source d'halogène actif.
3. Particule encapsulée selon la revendication 2 dans laquelle l'agent de blanchiment est un dichloroisocyanurate de métal alcalin.
4. Particule encapsulée selon la revendication 1 dans laquelle l'agent de séparation est une charge ou un adjuvant détergent soluble dans l'eau.

5. Particule encapsulée selon la revendication 4 caractérisé en ce que la charge ou l'adjuvant détergent est du sulfate de sodium, du chlorure de sodium, un phosphate condensé ou une combinaison de ces composés.
- 5 6. Particule encapsulée selon la revendication 5 dans laquelle l'éther cellulose soluble est une hydroxy (C₁₋₄) alkyl cellulose.
7. Particule encapsulée selon la revendication 6 dans laquelle l'hydroxy (C₁₋₄) alkyl cellulose est de l'hydroxypropylcellulose ou de l'hydroxyéthylcellulose.
- 10 8. Particule encapsulée selon la revendication 7 dans laquelle l'hydroxy (C₁₋₄) alkyl cellulose présente une DS de 0.7 à 3.0.
9. Particule encapsulée selon la revendication 8 dans laquelle l'hydroxypropyl cellulose présente une DS
15 de 1.4 à 3.0.
10. Particule encapsulée selon la revendication 8 dans laquelle l'hydroxy éthylcellulose présente une DS de 1.2 à 3.0.
- 20 11. Particule encapsulée selon la revendication 1 dans laquelle la particule encapsulée comprend 40 à 70% en poids de noyau, 10 à 50 % en poids de revêtement intérieur soluble dans l'eau et de 2 à 10 % en poids de revêtement extérieur.
12. Particule encapsulée selon la revendication 1, comprenant :
25 (a) 20 à 90 % en poids d'un noyau de source de chlore actif;
(b) 5 à 60 % en poids d'un revêtement intérieur soluble dans l'eau constitué d'une charge ou d'un adjuvant détergent enrobant le noyau et physiquement en contact avec celui-ci ; et
(c) 1 à 25 % en poids d'un revêtement extérieur encapsulant constitué d'une hydroxy (C₁₋₄) alkyl
30 cellulose qui est physiquement séparée du noyau de source de chlore actif par le revêtement intérieur.
13. Particule selon la revendication 12 dans laquelle la particule comprend :
(a) 40 à 70 % en poids de noyau;
(b) 10 à 50 % en poids d'un revêtement intérieur soluble dans l'eau constitué de sulfate de sodium,
35 de chlorure de sodium, d'un phosphate condensé ou d'une combinaison de ces composés ; et
(c) 2 à 10 % en poids d'un revêtement extérieur d'hydroxypropyl cellulose ou d'hydroxyéthyl
cellulose.

40

45

50

55